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FRETTING OF TITANIUM
AT TEMPERATURES
TO 650° C IN AIR

Robert C. Bill

Lewis Research Center

and U.S. Army Air Mobility R&D Laboratory

Cleveland, Obio 44135



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FRETTING OF TITANIUM AT TEMPERATURES TO 6500 C IN AIR

by Robert C. Bill

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SUMMARY

Fretting wear experiments were conducted on high-purity titanium at temperatures up to 650° C under a 1.47-newton normal load for fretting exposures of 3×10⁵ cycles duration in dry air. It was found that up to about 500°C, the fretting wear increased with temperature. Further increasing the temperature up to 650° C resulted in decreasing fretting wear. This behavior was explained in terms of a change in oxidation kinetics of titanium reported in the literature. At temperatures above 500° C the oxidation kinetics of titanium change from low rate cubic behavior to higher rate parabolic behavior. The oxide film can thus maintain a greater thickness and afford more wear protection at temperatures above 500° C. Further experiments were conducted at 650° C under conditions of varied normal load and number of fretting cycles. A transition from a low rate of fretting wear to a higher rate occurred after exposure to a number of fretting cycles that was dependent on the normal load. Scanning electron microscopy studies revealed that this transition was initiated by cracking and disruption of the oxide film. A model was proposed that coupled the oxidation rate kinetics of titanium at 650° C with the occurrence of wear at the surface of the oxide film. The model predicted that, under the heavier loads, considerable thinning of the oxide film should occur early in the fretting exposure. Under the lightly loaded condition, however, the thinning effect was not significant. It was further proposed that the observed cracking and disruption of the film and the transition to high wear was due to thinning of the oxide film to the extent that the load could no longer be supported by it.

INTRODUCTION

The presence of oxide films on metal surfaces plays an important role in the friction and wear behavior of metals. In many cases, the causes of significant transitions in wear rates have been identified with oxide film rupture (ref. 1), changes in the

structure and composition of surface oxides (refs. 2 to 4), and changes in oxidation rates. The role played by surface oxide films has been demonstrated to be as important in fretting wear situations as it is in other types of wear (refs. 5 to 7).

Experimental fretting observations reported in the literature (refs. 8 to 10), and some generalizing from sliding friction and wear observations suggest that metal fretting pairs may be divided into three categories based on dominant "steady-state" fretting wear mechanisms. The salient characteristics of the three categories are listed in table I. "Steady state" is meant to describe the situation after run in or after the initial Hertzian contact stresses have been relieved.

In the first category, fretting is controlled by the characteristics of the oxide film, and wear predominantly takes place within the film. This is the most desirable situation, as fretting is controlled by the properties of the oxide film, generally resulting in reduced wear. The catastrophic damage associated with the adhesive mechanism is effectively precluded. Examples include the high-temperature fretting of NiCrAl alloys (ref. 6), the preoxidized compressor blade damper wire experiments reported in reference 3, the fretting of various superalloys at elevated temperatures (ref. 11), and the fretting of film steel at temperatures above 200° C (ref. 7). The oxide films present in these cases resist spallation and are protective to the substrate metal from the standpoint of further oxidation.

A second category is one in which the surface oxide is completely worn away with each cycle of fretting motion. The oxide film that manages to form in the brief time interval between cyclic contact is not protective to the metal substrate either from the standpoint of mechanical damage or oxidation. The effect of the oxide may in fact be detrimental. Depending on the oxide properties, fretting wear of metals in this category can be aggravated by the abrasive action of oxidized debris. In reference 12 the higher fretting wear rate of titanium in air as compared to N₂ (room temperature experiments) was attributed to the abrasive effect of loose oxidized debris. Second-category-type behavior has been described in detail and modeled in reference 13. The model is supported by experimental results on mild steel. The wear equation proposed in reference 13 includes two terms, one of which accounts for mechanical damage (a modification of Archard's equation), the other accounting for the effect of oxidation rate.

The third category includes situations in which the surface oxide film is either absent (e.g., the noble metals, metals fretting in vacuum or inert environment) or is disrupted by the fretting action so that base metal to metal contact occurs. Oxidation plays no significant role in controlling the fretting process. The initial oxide film is not protective to the substrate from the standpoint of mechanical damage and the effects of loose oxidized debris are very secondary. The predominant fretting wear mechanisms are a combination of adhesion and fatigue, usually resulting in very high wear rates and extensive damage to the metal.

The purpose of this report is to characterize the high-temperature (650°C) fretting of titanium with particular reference to the role played by the surface oxide film. Titanium was chosen as the subject material for two reasons. First, the oxidation kinetics, which are key to the effectiveness of the oxide film at controlling wear, have been extensively studied and fairly well described at 650°C. Second, titanium is the basis for a number of alloys used in airframe and engine component applications. Its fretting characteristics are of special interest because they have a profound effect on the fatigue life of these components.

High-purity titanium was fretted at temperatures up to 650° C in dry air. The effect of normal load on fretting wear at 650° C was determined by conducting experiments with 0.294-, 0.735-, 1.47-, and 2.94-newton normal loads. At each load level, fretting exposures of about 3×10^4 , 10^5 , 3×10^5 and 10^6 cycles were applied. Wear volume measurements and scanning electron microscopy were used to evaluate the amount of fretting wear and the condition of the surface after fretting.

APPARATUS

A schematic diagram of the fretting rig is shown in figure 1, and a detailed description of its operation is included in reference 12. In essence, a linear oscillatory motion is provided by an electromagnetically driven vibrator with the frequency controlled by a variable oscillator. The load is applied to the specimens by placing precision weights on a pan which is hung from the load arm.

The fretting specimens consist of an upper, stationary, 4.76-millimeter-radius, hemispherical tip in contact with a lower flat which is driven by the vibrator.

During high-temperature experiments, the specimens and grip assemblies were surrounded by a 310-stainless-steel susceptor which was heated by an induction coil. The temperature was monitored by a thermocouple probe mounted in the lower grip. The grips were specially designed so that they stored sufficient elastic energy to ensure that no slippage would occur as a result of differential thermal expansion.

A dry-air environment was provided by flowing air through an absorption drier, then into the experimental chamber. In this way, moisture content was kept in the range of 10 to 100 parts per million.

MATERIALS

The titanium used in this investigation was of 99.8-percent purity. The principal impurities were carbon (150 ppm), oxygen (350 ppm), and silicon (150 ppm). In the as-

machined condition, the hardness of the specimens was 74 on the Rockwell B scale. After exposure to 650° C for 1 hour, the hardness was measured to be 69 (Rockwell B). Further exposure to 650° C conditions in air resulted in no further reduction in hardness.

PROCEDURE

Before fretting, the flat specimens were lapped, mechanically polished with 0.05-micrometer alumina polishing compound, then rinsed in distilled water. The hemispherical tips, ground to a 0.1-micrometer (4- μ in.) finish, were scrubbed with levigated alumina, and rinsed.

Next, a pair of like-metal specimens was assembled into the experimental chamber, which was then purged with dry air (or saturated air, if desired) for 1/2 hour before fretting. For the elevated-temperature experiments, the specimens were brought to the desired experiment temperature during the purbe by setting the plate current on the induction heater control so that the temperature stabilized at the desired level. After reaching the desired temperature, 1 hour was allowed for the apparatus to reach thermal equilibrium. With the vibrator now turned on, fretting was initiated by putting sufficient weight into the pan to just bring the specimens into contact (easily determined by a slight transmitted vibration). Additional weight was then added to apply the normal lead to the rubbing surfaces. Normal loads of 0.29, 0.74, 1.47, and 2.94 newsons were used. Other experimental conditions included a peak-to-peak amplitude of 70 micrometers (0.0027 in.), a fretting frequency of 80±0.2 hertz, and durations of 10⁶, 3×10⁵, 10⁵, and 3×10⁴ cycles.

Following each fretting experiment, the fretting scar on the flat surface was photographed to record the size and features of the wear scar and the debris accumulation around the scar. The loose debris was then rinsed off with commercial absolute ethyl alcohol, and a light section microscope was used to measure the wear volume on the flat specimen.

Specimens that were examined in the scanning electron microscope (SEM) were ultrasonically cleaned in methanol before viewing, to remove as much debris still adhering to the wear scar as possible.

RESULTS AND DISCUSSION

The results of the fretting wear volume against temperature experiment are shown in figure 2 for fretting exposures of 3×10^5 cycles under a 1.47-newton normal load in dry air. From room temperature up to about 450^0 C there is a trend of increasing fretting

wear with temperature. Somewhere between 450° and 540° C, the fretting wear volume goes through a maximum. At temperatures of 540° C and above, a trend of decreasing wear volume with increasing temperature is observed.

The increasing portion of the fretting wear against temperature curve (from room temperature to about 450° C) is most likely due to softening of the titanium. The thin oxide film that forms on the surface at temperatures up to about 450° C is disrupted because of the easy deformation of the substrate. This corresponds with fretting described under the third category presented in the INTRODUCTION.

At temperatures between 500° and 600° C, however, the oxidation kinetics of titanium undergo a change from a slow, cubic rate (proportional to the cube root of time) to a rapid parabolic one (refs. 14 and 15). This fast growing oxide film is able to maintain a greater thickness under fretting conditions, and is thus able to support the applied load without being disrupted. Evidence of this can be seen in figure 3. After 3×10^5 cycles under a 1.47-newton normal load at 450° C (fig. 3(a)), loose oxidized debris is seen around the wear scar. The surface of the wear scar itself is highly roughened indicating disruption of the oxide film and severe wear accompanying metal to metal contact. At 650° C, however, the oxide film was not penetrated after 3×10⁵ cycles under a 1.47newton normal load (fig. 3(b)). No loose debris is seen, and few features are visible in the wear scar. Even after 10⁶ cycles under the same conditions (fig. 3(c)), relatively little debris is seen and the wear scar surface is still quite smooth indicating that, at most, only localized oxide film penetration had occurred. Essentially, the fretting characteristics of titanium change from those described under category three to those under the first category, as discussed in the INTRODUCTION, when the temperature is increased to 650° C.

Effect of Load at 650° C

A detailed study of fretting wear at 650° C was undertaken to determine the oxide film durability under various applied normal loads. The results are shown in figure 4.

Examining the curves for the 0.74- and 1.47-newton normal loads in figure 4 makes it apparent that a transition to a high rate of fretting wear occurs after exposure to a number of cycles that is dependent on the normal load. The transition occurred at fewer than 3×10^4 cycles in the case of the 2.94-newton tests, while no transition is observed for 0.29 newton, at least up to 10^6 cycles.

The shape of the 1.47-newton curve suggests a periodic incidence of high wear rate alternating with periods of low wear rate. This can be understood in terms of the surface oxide film if penetration occurs periodically according to the following sequence:

(1) After a certain fretting exposure duration, localized film penetration occurs over a

small fraction of the wear scar, causing a high rate of wear. (2) As the area of heavy damage grows, the local contact stresses decrease and the wear rate attenuates. (3) Gradually, the oxide film can reform over the severely worn area; the contact load is redistributed; and a period of low wear occurs. The cycle might repeat itself a number of times before a truly steady-state situation is reached. Observe that the slope of the 2.94-newton curve is approximately parallel to the high wear rate portion of the 1.47-newton curve. This supports the contention that what is happening periodically under a 1.47-newton normal load occurs constantly under 2.94 newtons.

A transition from a low rate to a high rate of fretting was was observed under a 0.74-newton normal load after something more than 3×10^5 cycles. The change was much more slight than those seen under the higher normal loads though. No increase in wear rate was observed when the normal load was 0.29 newton, at least for fretting exposures of up to 10^6 cycles.

SEM Study of the Wear Scars

The wear scare resulting from fretting at 650° C were studied using scanning electron microscopy (SEM). A relation was sought between the features observed on the wear scars and the fretting exposure. Of particular interest was any change in these features that might coincide with the transition observed in the fretting wear rate.

The fretting wear scar resulting from 3×10^4 cycles under a 1.47-newton normal load is shown in figure 5. The features are typical of those seen in the early stages of fretting under all load conditions except the highest (2.94 N). Some signs of the surface distress resulting from the initial highly concentrated contact are still evident. In particular, the spall pits (0.5 to 1 μ m deep) and the areas of heavy plastic deformation are believed to be the product of low-cycle surface fatigue and early adhesive damage, respectively (refs. 12 and 16).

The surface of the wear scar shown in figure 6, after 1×10^5 fretting cycles under a 1.47-newton normal load, reveals none of the signs of severe surface disruption that were visible in figure 5. The spall pits and evidence of early plastic deformation have all been eradicated by the continued fretting action. Some fine cracks may be seen in the surface though. In general, the features observed after 3×10^4 cycles under a 2.94-newton load are similar to those of figure 6.

Exposure to 1×10⁵ cycles under a lighter load (0.74 N) resulted in the wear scar features shown in figure 7. Again, continued exposure to fretting has removed all evidence of the damage caused by the early concentrated contact. The dark patches in figure 7 indicate the areas where contact was occurring at the end of the fretting exposure. The oxide film is smoother and more compact than that on the surrounding noncontact surface. Note that the film is intact, supporting the full contact load.

The wear scar features typical of those seen early into the transition to accelerated wear are shown in figure 8 for the 1.47-newton normal load case. Cracks are present in the oxide film, with some of them apparently propagating into the substrate. It is significant that cracks were never observed in the most lightly loaded case (0.29 N), where no transition takes place, and that cracks were seen after only 3×10^4 cycles in the most heavily loaded case (2.94 N), where the transition must have occurred before 3×10^4 cycles according to figure 4.

As fretting continues beyond the transition, the wear scar features bear evidence that the oxide film becomes further disrupted. Referring to figure 9(a) reveals that, under the most heavily loaded condition (2.94-N normal load after 3×10^5 cycles), the entire wear scar surface has been heavily deformed and has an appearance reminiscent of wear scars generated at room temperature on titanium (ref. 12). The original oxide film has been completely broken up, and a new one of sufficient continuity and thickness to support the load is unable to form. Higher magnification (fig. 9(b)) reveals the presence of spall pits and details of the deformed load-carrying surface.

Under more lightly loaded conditions (1.47 and 0.74 N), the oxide film did not become disrupted over the entire wear scar surface. The severe damage, similar to that seen in figure 9, was confined to small regions occupying a fraction of the wear scar surface. Figure 10 (1.47-N load, 10^6 cycles) show a deep depression in the central portion of the wear scar with cracking in evidence at the bottom of the depression. This is where most of the fretting was occurring at the termination of the exposure. Adjacent to the depression is a region that appears to have undergone heavy surface damage with subsequent smoothing. The features that remain are similar to those of figure 9, with a plastically deformed, layered type of topography.

In summarizing the SEM study, features seen on the wear scar surfaces correlate with the transition to the high rate of wear. Prior to the transition, fretting proceeds as described under the first category (discussed in the INTRODUCTION). After the transition occurs, fretting locally proceeds according to the third category at those locations where the oxide film has been disrupted. The extent of film disruption over the wear scar surface seems to vary depending on load, being greater for higher normal loads. Surface cracks, initiated by oxide film rupture, marked the transition to rapid fretting wear. When these cracks were not observed (as in the 0.29-N case), fretting remained in the low wear mode (first category).

Interaction of Wear and Oxidation - A Model

It is hypothesized here that the oxide film breakup and transition to rapid fretting wear occurs after the film thickness has been reduced to a critical extent by the fretting action. At this point the contact load causes the film to crack. The cracks can quickly propagate into the substrate resulting in spallation and exposure of fresh metal surface. The fresh surface has but a thin oxide film when contact occurs there. Very quickly this film is broken up, and accelerated wear occurs.

The transition to accelerated wear may be predicted by combining a modified form of Archard's wear equation (ref. 17) with the oxide film growth equation. The wear (volume) rate is given by

$$\dot{V} = K_{OX} \frac{L}{P_{m}} fB \tag{1}$$

where \dot{V} is the wear rate, K_{ox} the wear coefficient of TiO_2 , P_m the hardness of titanium at 650° C, L the normal load, f the fretting frequency, and B the fretting amplitude. Note that equation (1) is meant to apply to the low wear rate fretting mode, before the transition occurs. There is precedent and justification for describing fretting wear with this equation. It is essentially the mechanical wear portion of the model proposed by Feng and Uhlig in reference 13. Also, the data in figure 4 show the fretting wear rate to be roughly constant when no transitions in wear mechanism occur, as in the case of the 0.294-newton normal load series. Furthermore, the fretting wear volumes measured after 3×10^4 cycles and 10^5 cycles are roughly proportional to the normal load for the 0.294-, 0.735-, and 1.47-newton curves.

The wear volume may be related to the idealized wear scar geometry (fig. 11) in the following way:

$$V \simeq 3.2 \text{ Rh}^2 - 1.6 \text{ h}^3$$

where R is the spherical radius of curvature of the wear scar surface, h is the maximum wear scar depth, and V is the instantaneous wear volume. Differentiating with respect to time gives

$$\dot{\mathbf{V}} = \frac{\partial \mathbf{V}}{\partial \mathbf{h}} \dot{\mathbf{h}} + \frac{\partial \mathbf{V}}{\partial \mathbf{R}} \dot{\mathbf{R}}$$

It was observed that R changes very little during the initial stages of fretting wear (between 3×10^4 and 3×10^5 cycles under all loads except 2.94 N). This is in agreement with general considerations of the fretting wear geometry of like-metal pairs (ref. 18). Hence, the second term on the right side is neglected, so

$$\dot{V} = \frac{\partial V}{\partial h}\dot{h} = 6.4 \text{ Rhh} - 4.8 \text{ h}^2\dot{h}$$

Since $h \ll R$, a further simplification is possible

$$\dot{\mathbf{V}} \doteq 6.4 \text{ Rhh}$$

Combining the previous expression with equation (1) results in

$$\dot{h} = \frac{K_{OX} \frac{L}{P_{m}} fB}{6.4 Rh}$$
 (2)

Equation (2) may be integrated to give

$$h = \left(\frac{K_{ox} \frac{L}{P_{m}} fB}{3.2 R}\right) t^{1/2} = C_{1} t^{1/2}$$

The oxidation of titanium at 650° C is observed to follow parabolic kinetics, based on weight increase measurements (refs. 14 and 15). The parabolic rate law may be expressed as

$$H = C_2^{1} t^{1/2}$$
 (3)

where H is the thickness of the oxide film, C is a rate constant, and t is the time of oxidation. Implicit in equation (3) is the assumption that the weight gain is proportional to the oxide film thickness. This is reasonable because, up to 800° C, the oxide film is composed exclusively of TiO_2 (rutile), with a defect concentration gradient across the film providing the driving force for the diffusion controlled growth. Equation (3) may be alternately expressed as

$$\dot{H} = \frac{(C_2')^2}{2H} = \frac{C_2}{H} \tag{4}$$

Note that, at any instant, the rate of increase in film thickness is inversely proportional to the thickness.

If the oxide film is undergoing wear while it is growing, the film thickness may be locally reduced. This results in an accelerated growth rate where there is wear. The situation is represented by the relation between h, G, and T in figure 11. The depth to

which oxidation has penetrated below the initial surface is given by G, the depth of the wear scar by h, and the thickness of the oxide film on the wear scar surface by T. The growth rate of the film \dot{G} with the simultaneous progression of wear is expressed by:

$$\dot{G} = \frac{C_2}{T} = \frac{C_2}{G - h} = \frac{C_2}{G - C_1 t^{1/2}}$$
 (5)

Equation (5) was solved numerically, and values of T were obtained as a function of time for cases representing high, intermediate, and low normal loads, determined by the value of C_1 . The results are shown in figure 12. The values chosen for C_1 were based on wear measurements made early in the 1.47-newton normal load series (before the transition to high wear). The value for the 1.47-newton case was doubled to simulate the high normal load, and halved to simulate the low normal load. The initial film thickness, before fretting, was on the order of 1 micrometer. Keep in mind that these results apply only so long as the oxide film remains intact, that is, fretting proceeds as under the first category. The significant point is that equation (5) predicts that the theoretical film thickness passes through a minimum value early in the fretting exposure under a high normal load, and no significant thinning is observed for the light load situation. Once disruption of the thinned oxide film occurs, equation (5) no longer describes the relation between oxide film growth and wear. The wear rate becomes much higher and the oxide film becomes very irregular and discontinuous.

The results may be generalized to other contact geometries. For conforming, or flat geometry cases, equation (1) predicts a uniform average rate of surface recession due to wear, as long as the oxide film is not ruptured. Film thinning will continue to the point at which, according to parabolic growth kinetics (eq. (4)), the oxide film growth rate can keep pace with wear. A steady-state film thickness is predicted. Film penetration may occur, leading to a transition to high wear, if the steady-state film thickness is insufficient to support the contact load.

CONCLUSIONS

High-purity titanium was subjected to fretting exposures of 3×10^5 cycles under a 1.47-newton normal load at temperatures up to 650° C. Fretting experiments were also conducted on high-purity titanium at 650° C under conditions of varied normal load and length of exposure. Based on the results of these experiments, the following conclusions are drawn:

- 1. The fretting wear increased with increasing temperature up to about 500° C. As the temperature increased beyond 500° C, fretting wear decreased. The change in trend with increasing temperature is attributed to a change in oxidation kinetics.
- 2. The experiments conducted at 650°C showed a transition from a low fretting wear rate to a higher one after exposure to a number of fretting cycles that was dependent on the normal load.
- 3. Scanning electron microscopy studies revealed that the transition was marked by cracking of the oxide film with eventual disruption of the film and heavy wear to the titatium as fretting continues.
- 4. Combining the wear rate equation with the equation describing the appropriate oxidation kinetics resulted in a fretting wear model that predicted a load dependent thinning of the oxide film. Thinning progressed until either the film could not support the normal load or until the oxidation kinetics could "overtake" the wear rate.

Lewis Research Center,

National Aeronautics and Space Administration, and

U.S. Army Air Mobility R&D Laboratory, Cleveland, Ohio, June 16, 1975, 505-04.

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TABLE I. - FRETTING WEAR CATEGORIES

Category	Name	Description			
I	Continuous protective oxide film	Oxide film Substrate			
П	Rapidly wearing oxide film	Loose oxide Interaction between loose oxide particles substrate and substrate			
ш	Metal-to-metal contact	Oxide film absent or disrupted in contact area			

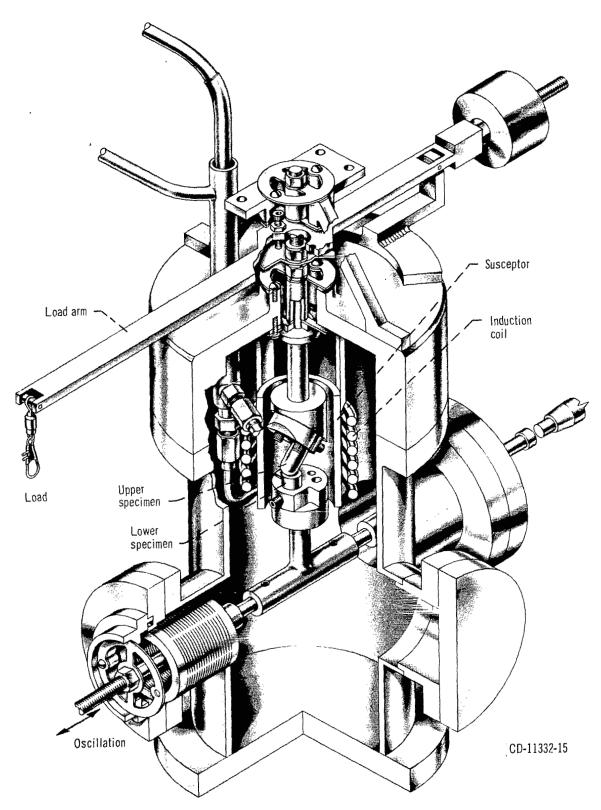


Figure 1. - Fretting apparatus.

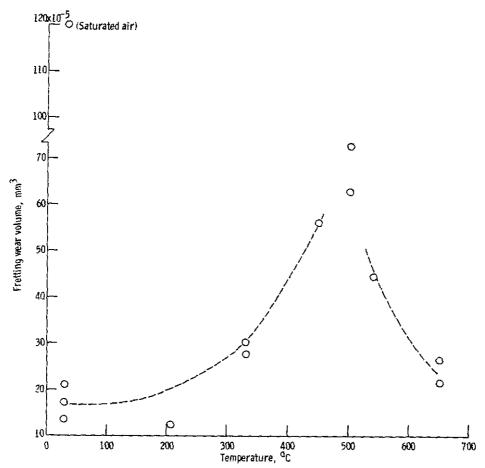


Figure 2. - Fretting wear volume plotted against temperature for high-purity titanium after $3x10^5$ fretting cycles under 1.47-newton normal load. Frequency, 80 hertz; dry air environment.



Figure 3. - Fretting wear scars on high-purity (itanium under 1.47-newton normal load. Frequency, 80 hertz; dry air environment; amplitude, 70 micrometers.

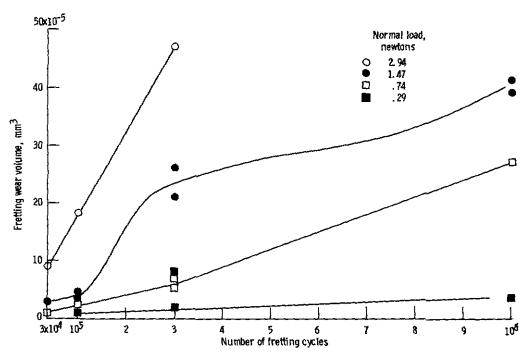
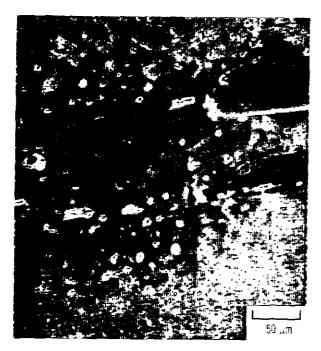


Figure 4. - Fretting wear plotted against number of fretting cycles for high-purity titanium at 650° C. Frequency, 80 hertz; dry air environment; amplitude, 70 micrometers.



(a) Overview.



(b) Enlargement of central region.



(c) Further enlargement showing heavy deformation and spalls.

Figure 5. - Scanning electron micrographs of wear scar on high-purity titanium after 3X104 cycles under 1.47-newton normal load at 650°C.

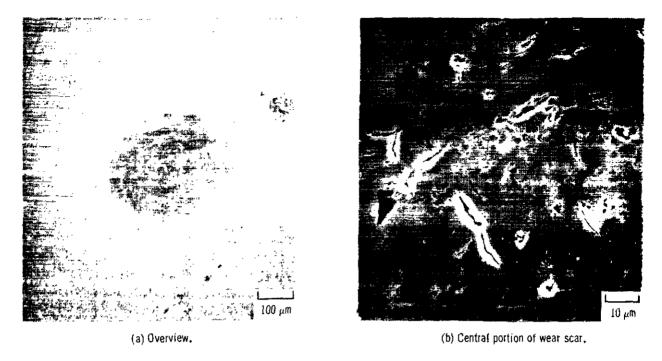


Figure 6. - Scanning electron micrograph of fretting wear scar on high-purity titanium after 1X10⁵ cycles under 1.47-newton normal load at 650 °C. Frequency, 80 hertz; amplitude, 70 micrometers.

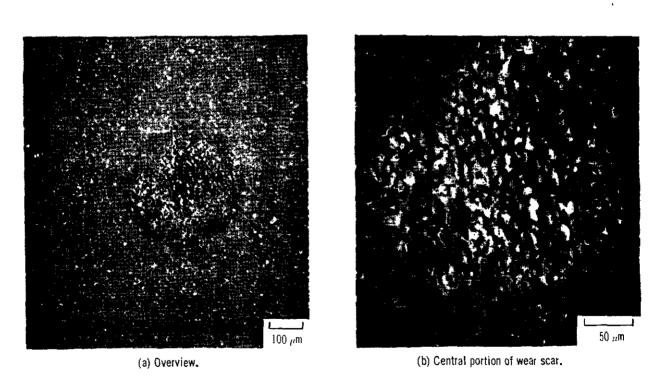
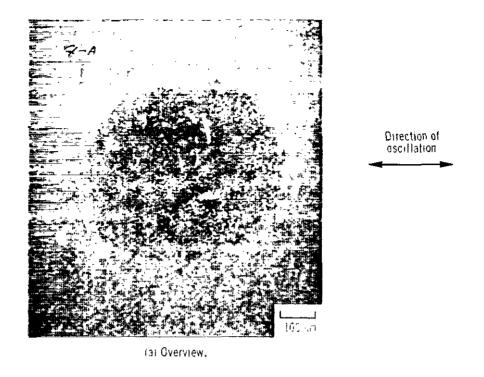
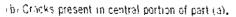
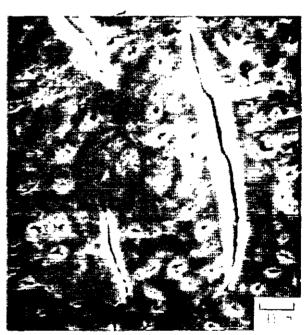


Figure 7. - Scanning electron micrograph of fretting scar on high-purity titanium after 3×10^5 cycles under 0.735-newton normal load at 650° C. Frequency, 80 hertz; amplitude, 70 micrometers.









(c) Cracks clearly observed in upper portion of wear scar.

Figure 8. - Scanning electron micrograph of fretting wear scar on high-purity titanium after 3X10⁵ cycles under 1.47-newton load at 650°C. Frequency, 80 hertz; amplitude, 70 micrometers.

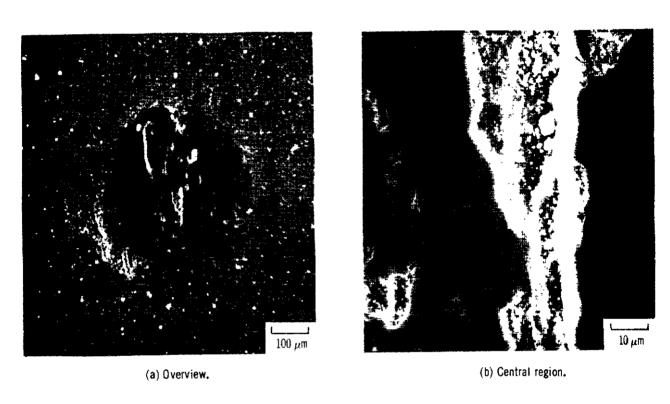


Figure 9. - Fretting wear scar on high-purity titanium after 3X10⁵ cycles under 2.94-newton normal load at 650°C. Frequency, 80 hertz; amplitude, 70 micrometers.

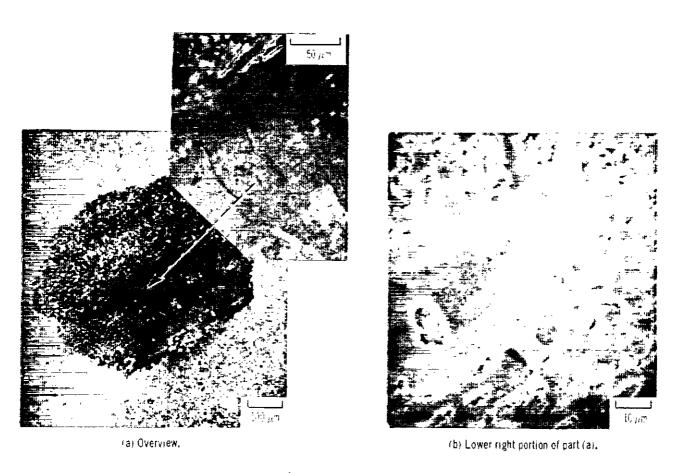


Figure 10. - Fretting wear scar on high-purity titanium after 106 cycles under 1.47-newton normal load at 650°C. Frequency, 80 hertz; amplitude, 70 micrometers.

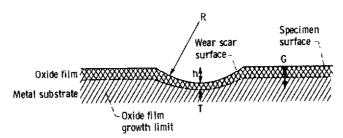


Figure 11. - Fretting wear scar section geometry.

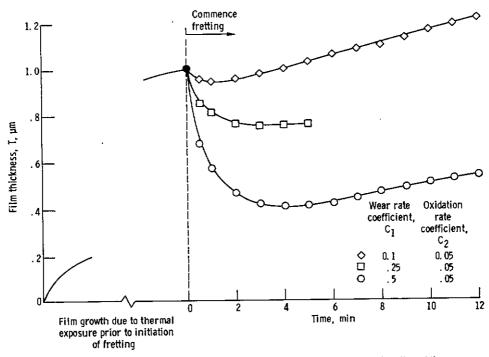


Figure 12. - Predicted oxide film thickness under fretting conditions as function of time.

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